PRELIMINARY EXPERIENCE WITH " IN-SITU " BAKING OF NIOBIUM CAVITIES *

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Abstract

In a series of experiments several single cell and multi-cell niobium cavities made from reactor grade and high RRR niobium (frequencies were 700 MHz, 1300 MHz and 1497 MHz) have been baked — after initial testing — "in-situ" around 145^0 C for up to 90 hrs prior to being recooled. Surprisingly, all cavities showed significant improvements in Q - values between 4.2 and 1.6K. The BCS surface resistance was lowered by nearly a factor of two. This cannot be explained by solely a reduction of dielectric losses caused by adsorbates at the surface or by a decrease of the mean free path due to possibly diffusion of oxygen into the surface layer. In several experiments also the high field behavior of the cavity improved after the "in-situ" baking procedure. The observed effect opens the possibility of significantly reducing the cryogenic losses for the CEBAF upgrade cavities, which in turn will permit to run the cavities at higher gradients if field emission loading can be prevented. Utilizing this effect can possibly translate into sizeable cost savings since fewer modules are needed for the upgrade program.

1 INTRODUCTION

Many experiments have been carried out over the years at different laboratories to investigate the contributions of oxide layers, adsorbates such as hydrocarbons or residual gas condensation during cooldown on the surface resistance of superconducting cavities. Systematic studies of the loss mechanisms caused by condensed gas layers on the cold cavity surfaces have been conducted by Piosczyk [1] with air, O_2 , N_2, CO_2 and N_2 /O_2 mixtures at 100 MHz. The experiments revealed that dielectric losses are occurring in these condensed layers, especially in gas species containing oxygen. In particular, condensed air has a loss tangent 60 times larger than pure oxygen. Room temperature exposure of cavities to air after initial testing resulted in significant Q-degradations [2]. This was confirmed in more recent experiments [3] at KEK, in which cavities exposed to air showed an increase in residual resistance, more pronounced multipacting and a degradation in maximum field gradient. However, an accumulated exposure for 3 days to dry nitrogen gas did not noticeably influence cavity behavior [4]. Another set of experiments showed that frozen-out gas layers—when thick enough—caused additional losses, but would not influence the field emission behavior if the surfaces were free of particulate contamination [5].

In a controlled study Palmer [6] investigated the contribution of surface oxides to the losses of a cavity. After initial heat treatment at 1400^0 C, which resulted in the dissolution of the natural surface oxides into the bulk, carefully regrown oxides contributed app. 1 - 2 n Ω to the surface resistance. Temperature maps taken in superfluid helium with sensitive thermometers showed large patches of increased losses besides localized defects [7]. Such patches can be chemical stains or residual contamination from inadequate final rinsing procedures. In summary, there is sufficient experimental evidence that dielectric losses can reduce the Q-values of superconducting cavities.

Nearly 10 years ago it was discovered that high purity niobium with a residual resistivity ratio (RRR) $\geq$ 250 is very sensitive to hydrogen precipitation at temperatures around 100K, resulting in Q-degradations of orders of magnitude for cavities made from such material [8]. The losses are uniformly distributed over the whole cavity surface; this "Q-disease" can be avoided by totally hydrogen degassing the material at temperatures T > 800^0 C or by fast cooldown through the dangerous temperature region of $70K \leq T \leq 130K$. Some significant reduction in "Q-disease" was also accomplished by "in-situ" diffusion of surface oxide into the bulk material at 250^0 C [9].

"In-situ" baking of electropolished cavities at $\approx 85^0$ C for $\geq$12 hrs has routinely been done at KEK, where the superiority of an electropolishing surface treatment over buffered chemical polishing was established in many experiments [10]; most intriguingly, severe Q-degradations at high gradients in the absence of field emission were not reported as was commonly observed with chemically treated cavities [11, 12].

Improvements of cavity performance by "in-situ" baking were reported more recently [13,14], especially at the higher gradient end of the Q_0 vs E_{acc} performance.

In the following we report on a series of experiments, during which—after initial testing—the cavities were baked "in-situ" for up to 90 hrs at a temperature of T $\approx$ 145^0 C and retested. To our surprise, significant reductions of the BCS surface resistance were observed,

which can not be explained by solely a reduction of dielectric losses caused by adsorbates or by a lowered mean free path caused by the diffusion of surface oxides into the bulk material.

In nearly every experiment also an improvement of the high field behavior of the cavities was observed, both with electropolished and chemically polished surfaces, consistent with observations made in other labs [15,16].

2 EXPERIMENTAL RESULTS

2.1 Experimental Procedure

The surface preparation of the cavities prior to the baseline tests was carried out by applying standard procedures: after degreasing of the cavities in a detergent with ultrasonic agitation, the cavity surface was chemically polished in a 1:1:1 - solution of hydrofluoric acid (48%), nitric acid (69%) and phosphoric acid (86%), subsequently rinsed with ultrapure water followed by a high pressure rinsing for 1 hr. The assembly of coupling probes was done in a class 100 clean room after a rinsing with reagent grade methanol; subsequently the cavities were attached to the test stand. Lately, the methanol rinsing step was eliminated after the cavity pumping system had been changed from an ion pump system to a 50 l/sec turbopump (integrated into the test set-up), backed by a scroll pump. The scroll forepump can easily handle the evacuation of "water-wet" systems and adds in combination with the small turbopump quite some convenience to the test preparation in addition to providing a better starting vacuum prior to cooldown.

The electropolishing of the cavities was done at KEK and they were shipped under vacuum to Jlab. In preparation for the tests only high pressure rinsing was applied to the surfaces.

After the baseline tests, during which the temperature dependence of the surface resistance was measured between 4.3K and 2K (in some tests to 1.6K) and the Q_0 vs E_{acc} behavior at 2K established, the cavities were warmed up to room temperature and subsequently baked in the dewar in a helium gas atmosphere surrounding the cavities. It took app. 24 hrs to reach the temperature of 145^0 C (just below the melting point of indium). During the baking the cavities were continuously pumped; the vacuum improved typically from the high 10^{-8} torr range to the low 10^{-8} torr range. The main gas species released during the baking were water, CO, CO_2 and methanol.

2.2. Experimental Observations

The following observations have been made during 17 experiments on 14 different niobium cavities, mainly baked at 145^0C, for durations from 10 hrs to 90 hrs :

- The BCS surface resistance decreases by up to a factor of two (depending on duration of baking, see figure 2) in the temperature range between 4.3K and 2K in high purity niobium cavities. The effect is much less pronounced in cavities fabricated from reactor grade niobium.

- The improvement is time and temperature dependent; it saturates after approximately 40 to 50 hrs.

- The value of Δ/kT_c generally increases slightly.

- The improvement in BCS surface resistance remains after air exposure or high pressure water rinsing.

- After the removal of approximately 3000 Å of material from the surface the original higher BCS surface resistance is restored; this increase in surface resistance is monotonic.

- The improvement is repeatable on any cavity.

- Sometimes the improvement in the BCS surface resistance was accompanied with an increase in residual resistance, offsetting the improvement. Longer baking times seemed to favor increases in residual resistance.

- In many cases an improvement of the high gradient performance of the cavities was observed, reflected in higher Q-values at increased gradients.

In the following, examples for the observations are given.

2.3 BCS Surface Resistance

Figure 1 shows a typical example of the effect of baking on the temperature dependence of the BCS surface resistance [the residual resistance is subtracted from the measured R(T)]. Over the whole temperature range of 4.3K≤ T ≤ 1.8K the BCS resistance of the 7-cell cavity JL7-1 is after baking for 35 hrs at 145^0 C below the baseline values. In addition, also a slight change in slope occurred.

In figure 2 the decrease of the BCS surface resistance at 4.3K is plotted as a function of baking time, based on the 17 independent tests carried out with 14 different cavities.

There is a clear exponential dependence, which saturates around 40 to 50 hrs. Because of this exponential dependence one can speculate that a diffusion process is involved in the change of the R_{BCS} ; most likely, oxygen from the interface is diffusing into the penetration depth of the superconducting material, thus modifying the material parameters [17].
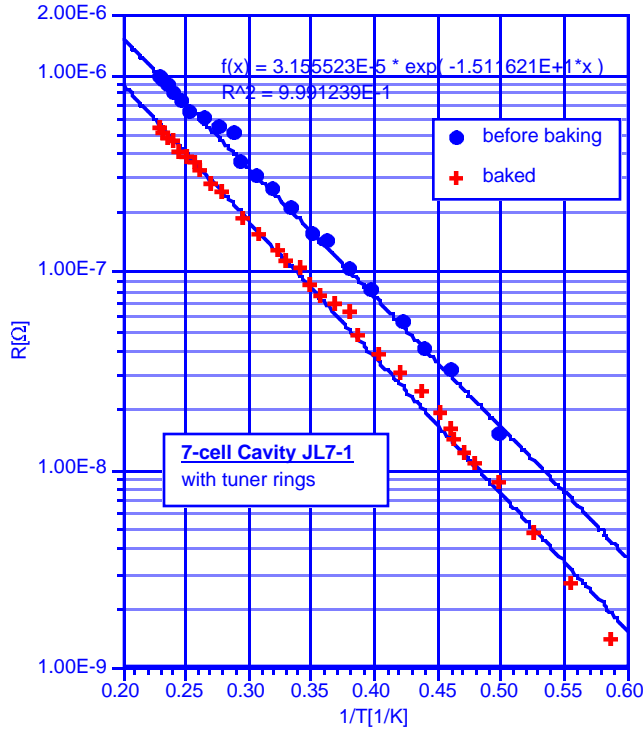
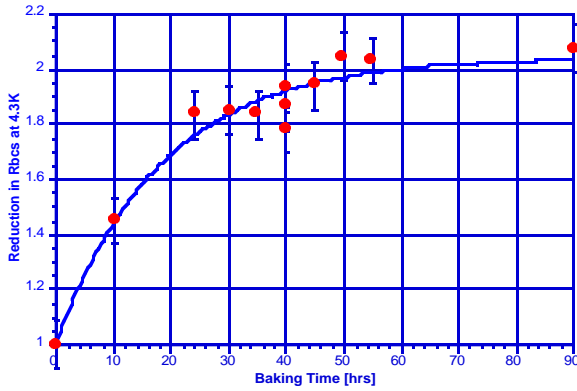Figure 1: Temperature dependence of the BCS surface resistance before and after "in-situ" baking at 145^0C.

Figure 2: Dependence of the reduction in BCS surface resistance at 4.3K on baking time at 145^0C.

In several exploratory experiments it had been established that the improvement of R_{BCS} after the baking was permanent as long as no material was removed from the surface. Neither "in-situ" exposure of the cavity surface to air (no disassembly of the test set-up) nor high pressure rinsing of the cavity after the initial experiment changed the lowered BCS surface resistance.

The remaining question of how deep the modified niobium surface extended into the bulk material after the baking was investigated with a set of tests, during which small amounts of material were removed successively from the surface by oxipolishing [18]: in this process the niobium surface is anodically oxidized in a solution of 30% NH_4OH at a low voltage (the voltage determines the thickness of the oxide layer, 20 Å/V) and after rinsing removed in hydrofluoric acid. Figure 3 shows the result of a set of 4 tests carried out with a single cell cavity.
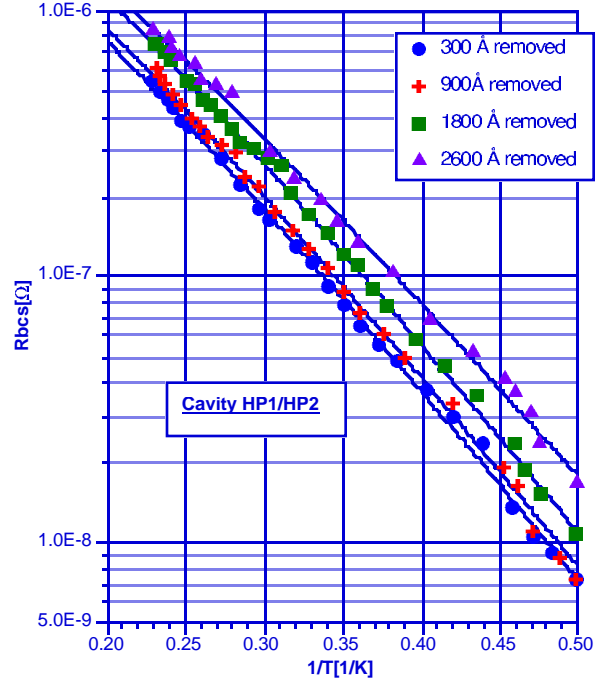

Figure 3: Temperature dependence of the BCS surface resistance after several steps of material removal by oxipolishing

Figure 4 then summarizes the "return" of the BCS surface resistance to its originally higher value as material is removed from the surface. After a removal of approximately 3000 Å of material the higher baseline resistance is restored.

A summary of the 17 experiments carried out with 14 different cavities is compiled in table 1. Only two tests were done with cavities made from reactor grade niobium (seamless cavities P3 and P4). In both cases only a rather small improvement of the BCS surface resistance in the range < 20 % was observed.

Cavity/ # of cells	Material/ Fabrication	Frequency [MHz]	Baking Time	Baking Temperature	R_{BCS} [4.3 K]	R_{BCS} [2 K]
P5-HAF Single cell	RRR/conv	1500	Baseline 10 hrs	140 °C	1×10^{-6} Ω 6.95×10^{-7} Ω	1.73×10^{-8} Ω 1.19×10^{-8} Ω
P4-1327 Single cell	RRR/conv	1500	Baseline (anodized) 40 hrs	145 °C	9.6×10^{-7} Ω 5×10^{-7} Ω	1.65×10^{-8} Ω 8.55×10^{-9} Ω
	RRR/conv	1500	HPR after baking		4.8×10^{-7} Ω	8.2×10^{-9} Ω
	RRR/conv	1500	2 min bcp 24 hrs	145 °C	9.1×10^{-7} Ω 5×10^{-7} Ω	1.4×10^{-8} Ω 5.3×10^{-9} Ω
HP1/HP2	RRR/conv	1500	Baseline 45 hrs	145 °C	9.7×10^{-7} Ω 5×10^{-7} Ω	1.43×10^{-8} Ω 4.8×10^{-9} Ω
	RRR/conv	1500	300 Å removed 900 Å removed 1800 Å removed 2600 Å removed		5.4×10^{-7} Ω 5.95×10^{-7} Ω 7.4×10^{-7} Ω 8.6×10^{-7} Ω	7.1×10^{-9} Ω 7.1×10^{-9} Ω 1.1×10^{-8} Ω 1.7×10^{-8} Ω
P5 Single cell	RRR/seamless	1500	Baseline 40 hrs	145 °C	9.8×10^{-7} Ω 5.3×10^{-7} Ω	1.5×10^{-8} Ω 7.7×10^{-9} Ω
P6 Single cell	RRR/seamless	1500	Baseline 55 hrs 90 hrs	145 °C 145 °C	1.1×10^{-6} Ω 5.2×10^{-7} Ω 5.1×10^{-7} Ω	2×10^{-8} Ω 7.8×10^{-9} Ω 7.4×10^{-9} Ω
	RRR/seamless	1500	e-polished 50 hrs	145 °C	8.8×10^{-7} Ω 4.3×10^{-7} Ω	1.14×10^{-8} Ω 5.3×10^{-9} Ω
P7 Single cell	RRR/seamless	1500	Baseline Test 30 hrs	140 °C	8.6×10^{-7} Ω 4.6×10^{-7} Ω	1.37×10^{-8} Ω 6.47×10^{-9} Ω
P3- Single cell	RG/seamless	1500	Baseline Test 40 hrs	145 °C	6.7×10^{-7} Ω 5.6×10^{-7} Ω	1.17×10^{-8} Ω 1×10^{-8} Ω
P4 Single cell	RG/seamless	1500	Baseline Test 40 hrs	145 °C	7.1×10^{-7} Ω 5.4×10^{-7} Ω	1.22×10^{-8} Ω 1.12×10^{-8} Ω
CEBAF 5/2 5-cell	RRR/conv	1500	Baseline 40 hrs	145 °C	8.1×10^{-7} Ω 4.6×10^{-7} Ω	1.33×10^{-8} Ω 6.1×10^{-9} Ω
JL 7-1 7-cell	RRR/conv	1500	Baseline Test 35 hrs	145 °C	9.6×10^{-7} Ω 5.3×10^{-7} Ω	1.46×10^{-8} Ω 8.8×10^{-9} Ω
K-15 Single cell	RRR/conv	1300	Baseline Test 50 hrs	145 °C	5.6×10^{-7} Ω 3.4×10^{-7} Ω	1.05×10^{-8} Ω 4.9×10^{-9} Ω
K-16 Single Cell	RRR/conv.	1300	Baseline 30 hrs	145 °C	5.4×10^{-7} Ω 3.7×10^{-7} Ω	1.15×10^{-8} Ω 7.9×10^{-9} Ω
JL-1 Single Cell	RRR/conv	1300	36 hrs 30 hrs	85 °C 145 °C	4.9×10^{-7} Ω 3.3×10^{-7} Ω	5.74×10^{-9} Ω 3.1×10^{-9} Ω
700- 1 3-cell	RRR/conv	700	Baseline 60 hrs	75 °C	2.22×10^{-7} Ω 1.79×10^{-7} Ω	2.8×10^{-9} Ω 1.96×10^{-9} Ω

Table 1: Summary of baking test; changes in BCS – surface resistances at 4.3K and 2K are shown

For two cavities (P4 -1327 and P6) the baking was done twice:

A). Cavity P4-1327 was tested with an anodic oxide layer of ≈ 400 Å during the baseline test, then baked; afterwards the cavity surface was chemically etched by ≈ 20 μm and after the second baseline test baked again. In both sets of tests the baking improved the BCS surface resistance quite reproducibly at 4.3K. At lower temperature (2K) the BCS surface resistances are different in both sets, indicating a change in the Δ/kT_c-value of the material. The anodic oxide layer in the first experiment was grown on the niobium surface for a twofold reason: first, if diffusion of oxygen into the penetration depth was responsible for the decrease in BCS surface resistance, additional oxygen might have a beneficial effect and promote further improvements and secondly, depletion of oxygen might have the visual effect of changing the color of the anodic oxide from dark blue (for a layer thickness of 400 Å) to another color. Neither a color change was observed nor was the BCS surface resistance lowered further due to the presence of the anodic oxide layer, indicating that effect

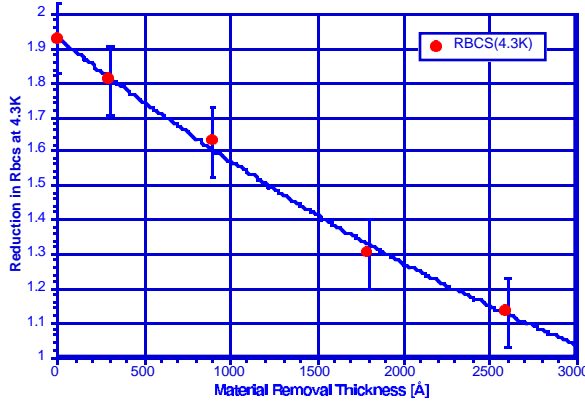caused by the "in-situ" baking is more subtle and might involve different properties of the interface [19,20]

Figure 4: Reduction in R_{BCS} due to material removal from the niobium surface after "in-situ" baking

B). The seamless cavity P6 was after the initial baseline test baked at 145^0 C for 55 hrs and subsequently another 35 hrs for a total of 90 hrs. The additional baking time did not further reduce the BCS surface resistance. This cavity was subsequently electropolished at KEK [17] by 40 μm and after high pressure rinsing at JLab retested again. The baseline test indicated a "wipe-out" of the baking effect from the previous test, even though the BCS surface resistance was somewhat lower (an effect, that is attributed to the electropolishing surface treatment [21]). A subsequent baking for 50 hrs resulted in the expected decrease in R_{BCS} enhanced by a change in Δ/kT_c.

In only two experiments was the baking temperature changed from 145^0 C to a lower temperature: single **cell cavity JL-1** was initially baked at 85^0 C for 36 hrs without a prior baseline test. However, the performance can directly be compared to cavities K-15 and K-16, which are of the same variety. In doing so, we realize that the 85^0 C baking already resulted in a app. 10% decrease in R_{BCS} at 4.3K; nevertheless, a much more dramatic change was accomplished by baking at the higher temperature of 145^0 C. Again, the experimental data at 2K vary in the different experiments as an indication of a change in Δ/kT_c. the **3-cell 700 MHz cavity 700-1** was after the baseline test baked at 75^0 C for 60 hrs and also exhibited already a small decrease in R_{BCS}. The baking experiment at the higher temperature is still pending.

2.4 High Field Behavior

As already mentioned in the introduction, in many cases an improvement of the high gradient behavior of the cavities was observed. Two examples are shown in figures 5 and 6 : figure 5 depicts the performance of the **electropolished** cavity K-15, which—after high pressure rinsing only—exhibited a severe multipacting

barrier at $E_{acc} \approx 27$ MV/m [3,22]. This degradation in Q-value disappeared after baking and the cavity performance improved both in Q-value and in gradient up to E_{acc}= 36 MV/m.

Figure 6 shows the performance of the seamless cavity P5 before and after baking. Only **buffered chemical surface treatment** had been applied to this cavity surface. The strong Q-degradation above $E_{acc} \approx 20$ MV/m in the baseline experiment prior to the baking is not only significantly reduced after the baking, but also a substantial increase in Q-value at 2K and an improvement in gradient to $E_{acc} \approx 33$ MV/m was achieved.

Halbritter [23] attributes the remarkable improvement of the high gradient behavior of the cavity to a reduction of interface losses, resulting in a higher Q-value at high gradients.
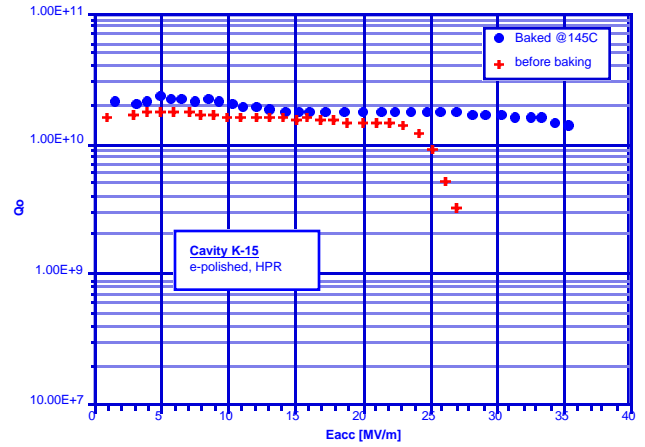

Figure 5: Q_0 vs E_{acc} for the electropolished cavity K- 15 before and after "in - situ" baking
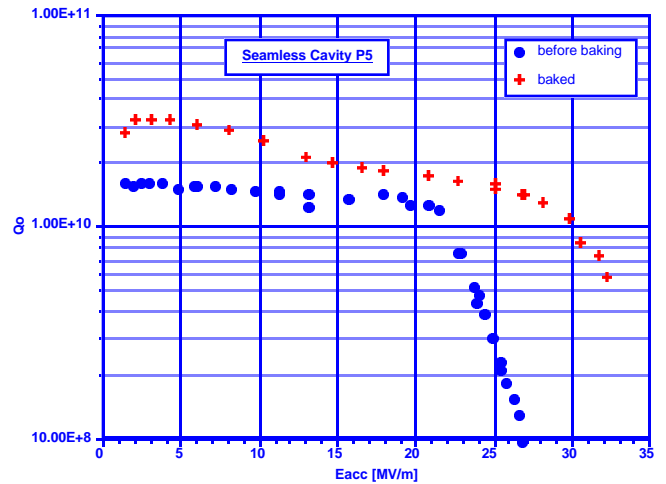

Figure 6: Q_0 vs E_{acc} for buffered chemically polished seamless cavity P5

Table 2 summarizes the experimental results of the baking tests with respect to the high gradient behavior of the tested cavities.

Cavity/ # of cells	Material/ Fabrication	Baking Time/ Temperature	$E_{acc, max}$ [MV/m]	Q_0 at $E_{acc, max}$	Comments
P4-1327 Single cell	RRR/conv	Baseline Test 45 hrs/145 °C	17.9 19.1	1.6×10^{10} 2.4×10^{10}	Quench Quench
HP1/HP2 Single cell	RRR/conv	Baseline Test 45 hrs/145 °C	21 25	3×10^9 1×10^{10}	Quench Quench
P5-HAF Single cell	RRR/conv	Baseline Test 10 hrs/140 °C	19.7 20.8	8×10^9 8.8×10^9	Quench Quench
P5 Single cell	RRR/seamless	Baseline Test 40 hrs/145 °C	26.8 32.5	1.3×10^9 6×10^9	Q-drop, power Quench
P6 Single cell	RRR/seamless	Baseline 45 hrs/145 °C	23.5 29.7	1×10^9 1.5×10^9	Q-drop, power Quench
P6 Single cell	RRR/seamless	Baseline Test 50 hrs/145 °C	32 31.2	2.3×10^9 8×10^9	e-pol. at KEK, HPR, Quench Quench
P7 Single cell	RRR/seamless	Baseline Test 30 hrs/140 °C	20.5 20.5	6×10^9 1×10^{10}	Quench Quench
K-15 Single cell	RRR/conv	Baseline Test 50 hrs/145 °C	27.3 35.6	3×10^9 1.3×10^{10}	e-pol.at KEK,HPR, MP barrier?
K-16 Single Cell	RRR/conv.	Baseline 30 hrs/145 °C	29.5 33	2.5×10^9 2.5×10^9	e-pol. at KEK, HPR, radiation Quench, radiation
JL-1 Single Cell	RRR/conv	36 hrs/85 °C 30 hrs/145 °C	29.5 29	7×10^9 8.3×10^9	e-pol + 85 ° C at KEK, HPR Quench
P4 Single cell	RG/seamless	Baseline 40 hrs/145 °C	15 15	1.6×10^{10} 1.6×10^{10}	Quench Quench
P3 Single cell	RG/seamless	Baseline 40 hrs/145 °C	16.2 15.5	8.2×10^9 5.8×10^9	Quench, radiation Quench, increased radiation

Table 2: Summary of baking test; change in high field behavior is listed

3   DISCUSSION OF POTENTIAL BENEFITS OF BAKING

As shown above, the "in-situ" baking effect can improve both the Q-value and the gradient of a niobium cavity made from high purity niobium. For the CEBAF upgrade project, where only limited refrigeration power is available, which limits the useable gradients to $E_{acc} \approx 12.5$ MV/m at a Q-value of $Q = 6.5 \times 10^9$, the utilization of this effect might have a twofold benefit: first, it might be possible to operate the upgrade cavities at a higher gradient and secondly, they might be operated at a higher temperature close to $T\lambda$. In both cases cost savings seem possible.

To verify these potential benefits we want to answer three questions:

A). At what level of residual resistance R_{res} is the decrease in BCS surface resistance due to baking compensated, wiping out all improvements?

Based on the experimentally achieved BCS-values the Q - value

$$Q = G/(R_{BCS} + R_{res})$$

(G = geometry factor $\approx 275 \ \Omega$) is plotted as a function of R_{res} for the baseline and the "in-situ" baking case in figure 7 for the two temperatures of 2K and 2.17K. First one notices that of course high residual resistances will dominate the achievable Q-values and that residual resistances above $\approx 20 \ n\Omega$ are not desirable. Secondly however, figure 7 shows that after baking the Q-values at 2.17K are slightly higher than the Q-values at 2K for the unbaked case: therefore the baking effect gives the potential of raising the operation temperature of cavities

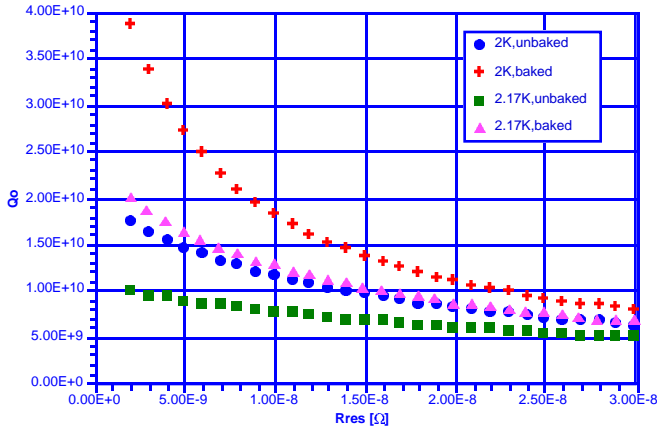to 2.17K with the same cavity Q-values or cryogenic losses, respectively.

Figure 7: Calculated Q_0-values for unbaked and baked CEBAF upgrade cavities as a function of R_{res}

B). Which residual resistance can one "afford" for an upgrade 7-cell CEBAF cavity at a gradient of $E_{acc} = 12.5$ MV/m, if a cryogenic capacity of 17.5 Watts per cavity is available?

Based on the experimentally achieved R_{BCS} values the dissipated power in the cavity walls of a 7-cell upgrade cavity is plotted vs R_{res} in figure 8 for two different temperatures (2K and 2.17K) and for the two cases of unbaked and baked cavities. One can see again that after baking the losses at 2.17K are still slightly less than without baking at 2K. For losses of 17.5 W, a residual resistance of $R_{res} \approx 30$ nΩ is "affordable" at 2K, unbaked and at 2.17 K, baked, whereas for a 2.17K operation of an unbaked cavity the residual resistance has to be ≤ 16 nΩ. A baked cavity can still be operated at 2K with $R_{res} \approx 35$ nΩ.
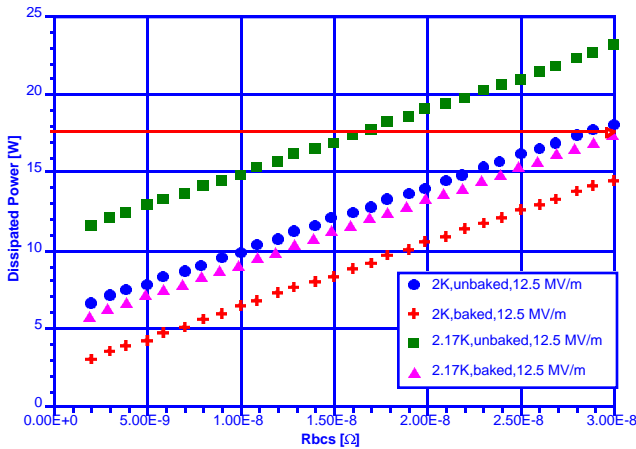

Figure 8: Calculated power dissipation for a 7-cell cavity at $E_{acc} = 12.5$ MV/m and at 2K and 2.17K before and after baking (based on measured R_{BCS} − values)

C). At what gradients and what values of R_{res} can a baked upgrade cavity be operated at 2.17K ?

Based on the experimentally achieved R_{BCS}-values after baking the dissipated power in the cavity walls of a 7-cell upgrade cavity at 2.17K is plotted vs R_{res} for different gradients in figure 9. One can see that a gradient of $E_{acc} \approx 18$ MV/m will require a residual resistance of $R_{res} \approx 10$ nΩ to meet the power dissipation limit of ≈ 17.5 Watts. Such R_{res}-values can be achieved rather routinely, if field emission loading is avoided.
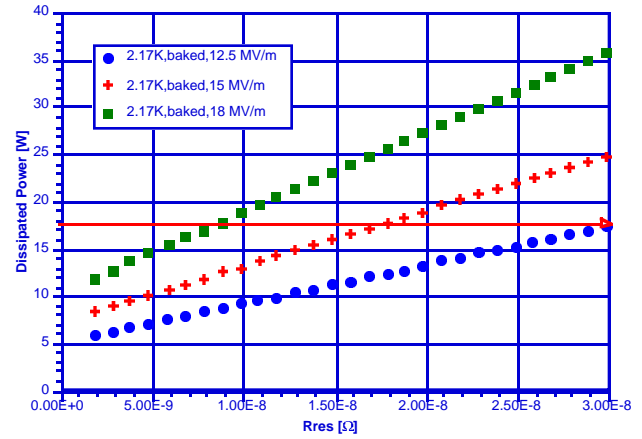

Figure 9: Power dissipation for a baked 7-cell upgrade cavity at 2.17K, based on measured R_{BCS} - values for different E_{acc}

4 SUMMARY

"In-situ" baking of niobium cavities at moderate temperatures around 145^0 C for extended periods of time can significantly improve the performance of these cavities. The BCS surface resistance is reduced by roughly a factor of 2 and in many cases also an improvement of the high gradient behavior was observed. The lowering of the BCS surface resistance cannot solely be explained by a mean free path effect, but other material parameters such as Δ/kT_c and the penetration depth seem to change also. The improvement in R_{BCS} disappears only after the removal of several thousand Ångstom of material from the surface. Most likely the change in material parameters is caused by diffusion of surface oxides/suboxides into the material [17].

This "in-situ" baking effect might have a twofold benefit for the CEBAF upgrade project, where only limited refrigeration power is available: first, it might be possible to operate the upgrade cavities at higher gradients as originally anticipated and secondly it might be possible

to operate them at a higher temperature close to $T\lambda$. In both cases cost savings are possible.

Because of the ease of the "in-situ" baking this step should become a routine procedural step in the preparation of a niobium cavity in the same way as chemical surface treatment and high pressure ultrapure water rinsing are routinely used. However, as mentioned earlier, an increase in residual resistance was sometimes observed in these experiments, which can offset the gains from the baking. It is not yet clear under which experimental conditions the increase in R_{res} occurs—prolonged baking times seem to favor this change— and further investigations are needed to avoid this disturbance and to take full advantage of the performance improvements due to the "in-situ" baking.

5 ACKNOWLEDGEMENT

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